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Indian Standard

METHODS OF SAMPLING AND TEST FOR FERTILIZERS

PART 4 DETERMINATION OF POTASSIUM

(*First Revision*)

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July 1985

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Indian Standard

METHODS OF SAMPLING AND TEST FOR FERTILIZERS

PART 4 DETERMINATION OF POTASSIUM

(First Revision)

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Indian Standard

METHODS OF SAMPLING AND TEST FOR FERTILIZERS

PART 4 DETERMINATION OF POTASSIUM

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 4) (First Revision) was adopted by the Indian Standards Institution on 29 January 1985, after the draft finalized by the Fertilizers Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1971. Taking into consideration the views of the manufacturers, testing authorities and the technologists, the Sectional Committee decided to revise this standard and felt that the methods of test should be related to those routinely used in the industrial and fertilizer testing laboratories.

0.3 In the present revision the 'sodium tetraphenylboron (STPB)' volumetric method is given as a referee method and sodium tetraphenylboron 'gravimetric' method as an alternate referee method and shall be used in case of any dispute. The other two methods, namely, 'perchloric acid' and 'flame photometric methods' shall be used as alternate methods.

0.4 In order to facilitate cross-reference to the present revised standard [IS : 6092 (Part 4) (First Revision)] with the original standard [IS : 6092 (Part 4) - 1971], Appendix A has been included in this standard showing the corresponding methods of test.

0.5 In the preparation of this standard, consideration has been given to the need for maintaining co-ordination with the methods of sampling and test for fertilizers prescribed under the *Fertilizer* (*Control*) *Order*, 1957, and the *Essential Commodities Act*, 1955 (incorporating amendment up to August 1981) of the Government of India.

0.6 In the preparation of this standard, assistance has been derived from the following publications:

Official methods of analysis. 1975. Association of Official Analytical Chemists, Washington DC, USA.

Recommended analytical methods. National Plant Food Institute, Washington DC.

The fertilizers and feeding staff regulations 1960. Department of Agriculture, UK.

0.7 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 4) prescribes the methods for determination of potassium in its various forms in fertilizers, including fertilizer mixtures.

2. SAMPLING

2.1 Representative test samples of the fertilizer shall be prepared as prescribed in IS : 6092 (Part 1)-1985†.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977‡) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. PREPARATION OF SAMPLE FOR ANALYSIS

4.1 Preparation of sample for analysis shall be done as prescribed in IS : 6092 (Part 1)-1985†.

5. METHODS

5.1 Three methods for determination of potassium are given in this standard. The method given in **6** is the referee method and shall be used in case of any dispute. The methods given in **7** is an alternate reference method and the methods given in **8** and **9** shall be used as alternate methods depending upon the availability of apparatus and reagents.

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*Rules for rounding off numerical values (*revised*).

†Methods of sampling and test for fertilizers: Part 1 Sampling (*first revision*).

‡Specification for water for general laboratory use (*second revision*).

6. SODIUM TETRAPHENYLBORON VOLUMETRIC METHOD

6.0 General — This method is applicable to both mixed and straight potassium fertilizers.

6.1 Reagents

6.1.1 *Sodium Hydroxide Solution* — 20 percent. Dissolve 20 g sodium hydroxide pellets in about 80 ml of water, allow to cool and make up to 100 ml.

6.1.2 *Formaldehyde Solution* — 35 to 37 percent.

6.1.3 *Sodium Tetraphenylboron (STPB) Solution* — approximately 1·2 percent. Dissolve 12 g of sodium tetraphenylboron in approximately 800 ml of water. Add 20 to 25 g of aluminium hydroxides. Stir for 5 minutes, and filter through Whatman No. 42 or equivalent into one litre volumetric flask. Rinse the beaker with small portions of water and add through the filter paper to the filtrate. Add 2 ml of 20 percent sodium hydroxide solution to the filtrate, mix and dilute to volume with water. Let it stand for 48 h and then standardize, adjust the concentration so that 1 ml of the solution is equivalent to 1 percent K_2O. Store at room temperature away from light.

6.1.4 *Benzalkonium Chloride (BAC) Solution* — approximately 0·625 percent. Dilute 50 ml of 12·8 percent benzalkonium chloride to one litre with water, mix and standardize. Cetyl trimethyl ammonium bromide may be used instead of benzalkonium chloride. If another concentration is used then adjust the concentration of this solution so that 2 ml is equal to 1 ml of STPB.

6.1.5 *Clayton Yellow (Titan Yellow) Indicator* — Dissolve 0·1 g of clayton yellow in 250 ml of water.

6.1.6 *Ammonium Oxalate* — 4 percent. Dissolve 40 g of ammonium oxalate monohydrate in water and dilute to one litre.

6.1.7 *Potassium Dihydrogen Phosphate (Reagent Grade)* — Dry at 105°C for two hours.

6.2 Procedure for Standardization of Solutions

6.2.1 *Benzalkonium Chloride (BAC)* — In a 125 ml volumetric flask, add 1 ml of 20 percent sodium hydroxide solution, 2·5 ml formaldehyde solution, 1·5 ml of 4 percent ammonium oxalate solution, 1·0 ml of STPB solution (from burette), mix well, dilute with 20 to 25 ml water and add 6 to 8 drops of clayton yellow indicator. Titrate to pink end point with BAC solution, using 10 ml semi-micro burette, and then dilute this reagent to the proper concentration. (A suitable working concentration shall be 1 ml of STPB = 2 ml of BAC.)

6.2.2 *Sodium Tetraphenylboron Solution* — Dissolve 2·5 g of potassium dihydrogen phosphate in about 100 ml of water in a 250 ml volumetric flask, add 50 ml of 4 percent ammonium oxalate solution, dilute to volume with water and mix. Transfer 15 ml aliquot (equivalent to 51·9 mg K_2O) or 43·10 mg K to a 100 ml volumetric flask, add 2 ml of 20 percent sodium hydroxide solution, 5 ml of formaldehyde solution, and 43 ml of STPB reagent from burette, dilute to volume with water, mix thoroughly, let it stand for 10 min and pass through a dry filter. Reject the first few ml of filtrate and transfer 50 ml to a 150 ml conical flask, add 6 to 8 drops of clayton yellow indicator and titrate excess reagent with BAC solution to the pink end point.

Calculate titration value as follows:

a) $$F = \frac{34{\cdot}61}{43\text{ ml} - \text{ml BAC solution}} = \text{percent } K_2O/\text{ml of STPB reagent}$$

or

b) $$F = \frac{28{\cdot}73}{43\text{ ml} - \text{ml of BAC solution}} = \text{percent K/ml of STPB reagent}$$

The factor 'F' applies to all test materials if 2·5 g sample is taken and diluted to 250 ml and 15 ml aliquot is used for titration. If this factor 'F' is to be calculated in terms of mg K_2O/ml STPB reagent, then following calculations may be followed:

$$F = \frac{51{\cdot}92}{43\text{ ml} - 2 \times \text{ml of BAC solution} \times A} = \text{mg } K_2O/\text{ml STPB}$$

where

A = STPB equivalent to 1 ml of BAC.

6.3 Preparation of Solution

6.3.1 *Mixed Fertilizers* — Take accurately weighed 2·5 g of prepared sample (1·25 g if K_2O is more than 50 percent) in 250 ml conical flask. Add 125 ml of water in 50 ml of 4 percent of ammonium oxalate solution. One millilitre of diglycol stearate solution may be added if needed to prevent foaming. Boil for 30 min; and slight excess of ammonium hydroxide and after cooling, make up to a volume to 250 ml in a volumetric flask with water, mix well and pass through a dry filter paper.

6.3.2 *Potassium Salts (Potassium Chloride, Potassium Sulphate, Potassium Magnesium Sulphate and Mixed Potash Salts)* — Dissolve 2·5 g (1·25 g if K_2O is more than 50 percent) of the sample in water and dilute to 250 ml without adding ammonium oxalate and ammonium hydroxide. When interferring substances, such as calcium and aluminium are present, proceed as in **6.3.1**.

6.4 Procedure

6.4.1 Transfer 15 ml aliquot from 6·3 to a 100 ml volumetric flask, add 2 ml of sodium hydroxide solution and 5 ml of formaldehyde solution. Mix by swirling and let it stand for 5 min. Add 1 ml of standard STPB solution for each 1 percent of K_2O expected in the aliquot and an additional 8 ml excess to ensure complete precipitation. Do not swirl or mix while STPB is being added. Dilute to mark with water, mix thoroughly. Let it stand for 10 min and pass through dry filter paper. Discard first few ml of the filtrate.

6.4.2 Transfer 50 ml aliquot of the filtrate to 150 ml conical flask. Add 6 to 8 drops of Clayton Yellow indicator and titrate the excess reagent with standard BAC solution to the end point using 10 ml micro-burette.

6.4.2.1 *Calculation*

$$K_2O \text{ percent } (M/M) = \frac{(X - 2 \times V_2 \times A) \times F \times D}{10 \times M}$$

where

X = volume ml of STPB added to sample aliquot in **6.4.1**;

V_2 = volume ml of BAC solution required for the titration in **6.4.2**;

A = STPB equivalent to 1 ml of BAC (obtained in **6.2.1**);

F = mass in mg of K_2O equivalent to 1 ml of STPB (obtained in **6.2.2**);

M = mass in g of sample in the aliquot taken; and

D = total dilution done = $\frac{250}{15}$.

7. SODIUM TETRAPHENYLBORON GRAVIMETRIC METHOD (ALTERNATE REFERENCE METHOD)

7.0 General — The procedure is based on the precipitation of potassium in test solution with STPB in a weakly alkaline medium in presence of EDTA, disodium salt (disodium ethylene diamine tetraacetate dihydrate) and formaldehyde solution to check interference by polyvalent and ammonium ions. The method is suitable for determination of potassium content of fertilizers (straight and mixtures) but not applicable in presence of urea, cyanamide and organic materials which should be destroyed by pre-treatment (dry ashing, boiling with alkali, etc) as given under **7.2**.

7.1 Reagents

7.1.1 Sodium tetraphenylboron (approximately 1·5 percent). Dissolve 7·5 g of STPB in 475 ml of water. Add 4 ml of 20 percent sodium hydroxide and 20 ml of 10 percent solution of magnesium chloride hexahydrate. Stir well for 10-15 min and filter through a fine textured filter paper. The solution shall be stored in dark in polythene bottles for 4-5 weeks and should be filtered before use.

7.1.1.1 *STPB wash solution* — Dilute one volume of the STPB solution with 10 volumes of water.

7.1.2 *EDTA Disodium Salt Solution (4 Percent)* — Dissolve 4·0 g of the pure salt in water and make up to 100 ml.

7.1.3 *Magnesium Chloride Solution (10 Percent)* — 10 g of $MgCL_2 \cdot 6H_2O$ dissolved in water and made up to 100 ml.

7.1.4 *Formaldehyde Solution (30 Percent)* — Filter if needed before use.

7.1.5 *Phenolphthalein Indicator* — 0·5 g of phenolphthalein dissolved in 100 ml of 95 percent ethyl alcohol.

7.2 Procedure — Transfer with a pipette a suitable aliquot of the test solution containing 30 to 50 mg of K_2O to a 250 ml beaker and adjust the volume to about 50 ml by evaporation or dilution. To this, add 20 ml of EDTA solution and few drops of phenolphthalein indicator. Add drop by drop the sodium hydroxide solution for the red colour to appear and then add 2 ml of this in excess. Boil for 15 min.

NOTE — Too high concentration of sodium hydroxide may cause precipitation of ferric iron (if present in the test solution) and coprecipitation of STPB, during boiling.

Run down 10 ml of formaldehyde solution and if necessary a few drops of sodium hydroxide solution until the red colour persists. Cover the beaker with a watch glass and keep on a steam bath for about 15 min. The solution should remain red. If necessary add the NaOH solution drop by drop to restore the red colour.

Remove the beaker from the steam bath and add immediately drop by drop while stirring 40 ml of STPB solution. Stir for 2 min more and cool rapidly to below 20°C in running or iced water and allow to stand for 10 min.

Take a previously dried (in oven at 120°C) and weighed, filter crucible (sintered glass or porcelain disc of porosity P_{20}) fitted with a mild suction and decant the supernatant liquid through it. Wash the precipitate in the beaker with 40 ml of wash solution and decant again and repeat this procedure. Transfer the precipitate quantitatively, rinse with two 20 ml portions of wash liquid followed by 5 ml of cold water. Dry the crucible with precipitate in oven at 120°C for 1½ h, allow to cool in desiccator and weigh again. Carry out a blank test at the same time.

7.3 Calculations

Potassium content, expressed as K or K_2O as percentage by mass

$$= \frac{[(M_2 - M_1) - (M_4 - M_3)] \times F \times V_0 \times 100}{M_0 \times V_1}$$

where

M_0 = mass in g of the test portion of the material;
M_1 = mass in g of the dry filter crucible;
M_2 = mass in g of crucible plus precipitate (dry);
M_3 = mass in g of the crucible used for blank;
M_4 = mass in g of the crucible in blank test with precipitate (if any);
V_0 = volume in ml of the test solution (volume finally made up);
V_1 = volume in ml of the aliquot of the test solution taken for the precipitation; and
F = factor, if expressed as K = 0·109 1, if as K_2O = 0·131 4.

8. PERCHLORIC ACID METHOD

8.0 General — This method depends on the insolubility of potassium perchlorate and the solubility of sodium perchlorate in alcohol, and is applicable in presence of alkali metals, chlorides and nitrates. Sulphates and ammonium salts must be absent on account of the low solubility of sodium sulphate and of ammonium perchlorate in alcohol. Phosphates, organic matter, etc, shall be removed. Methods are given for the elimination of the interferring substances.

8.1 Reagents

8.1.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

8.1.2 *Barium Chloride Solution* — Dissolve 100 g of crystalline (reagent grade) barium chloride in water, filter the solution if necessary and dilute to one litre.

8.1.3 *Dilute Hydrochloric Acid* — Dilute 200 ml of concentrated hydrochloric acid with water to one litre.

8.1.4 *Calcium Oxide* — finely ground.

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*Specification for hydrochloric acid (*second revision*).

8.1.5 *Ammonium Hydroxide Solution* — Specific gravity (0·88-0·89).

8.1.6 *Ammonium Carbonate Solution* — saturated aqueous solution.

8.1.7 *Ammonium Oxalate Solution* — saturated aqueous solution.

8.1.8 *Perchloric Acid Solution* — 20 percent (60-70 percent material may be suitably diluted). This solution should not contain any interferring materials (*see* **8.0**).

8.1.9 *Alcohol* — Rectified spirit, 95 to 96 percent (*v/v*). May be distilled, if necessary.

8.1.10 *Wash Solution* — Add potassium perchlorate to alcohol and shake until a saturated solution is obtained. Keep the solution over solid potassium perchlorate and filter immediately before use.

8.2 Preparation of Sample Solution

8.2.1 *For Potassium Salts Free from Sulphates and Other Interferring Substances* — Weigh out the test material accurately to the nearest mg an amount equivalent to potassium content of 1·5 to 2·0 g of (K_2O). Dissolve in water; dilute to 500 ml in a volumetric flask, mix well and filter through a dry filter. Reject the first few portions of the filtrate. Determine the potash in 50 ml of solution by precipitating with perchloric acid as described under **8.3**.

8.2.2 *For Potassium Salts Containing Sulphates and Other Interferring Substances But Free from Organic Matter* — This procedure is given for eliminating the interference caused by presence of sulphate. If the sample contains phosphates, iron, manganese or substances other than sulphate which interfere wiih the determination of potash, the method described for mixed fertilizers (**8.2.3**) should be used.

Weigh accurately a portion of the sample equivalent in potassium content to 1·5 to 2·0 g of K_2O into a 500-ml beaker. Add about 300 ml of water and 20 ml of concentrated hydrochloric acid and heat the solution to boiling. To the boiling solution add cautiously, drop by drop, barium chloride solution in an amount slightly in excess of that necessary to ensure the complete precipitation of sulphate. Cover with a watch glass and keep on a water bath for about half an hour so that precipitate settles below. A thin stream of barium chloride solution to the supernatant liquid should not create any turbidity to ensure total precipitation of sulphate. Cool the liquid to about 20°C, transfer to a 500-ml volumetric flask, dilute to mark, mix and filter through a fine texture dry filter. Take 50 ml of the filtrate and evaporate to dryness in a basin and moisten the residue with concentrated hydrochloric acid. Again evaporate to dryness, dissolve the residue in 5 to 10 ml of dilute hydrochloric acid and filter if necessary collecting all the washings (till free from chloride) along with the filtrate. Determine the potash in the solution by the method described under **8.3**.

8.2.3 *For Potassium in Mixed Fertilizers*

8.2.3.1 Weigh accurately about 10 g of the sample, If organic matter is present, gently incinerate preferably in a muffle furnace at a temperature not exceeding 500°C. Transfer the weighed portion of the sample or the incinerated residue to a 500 ml beaker with a little water, add 10 ml of concentrated hydrochloric acid and then warm for ten minutes. Dilute with water to about 300 ml and bring gradually to boiling point. Add 10 g of calcium oxide made into a paste with water. Bring the contents again gently to the boiling point, and keep this heated for about half an hour with frequent stirring. Cool to room temperature, transfer to a 500 ml volumetric flask, dilute to mark and, after thorough shaking, filter through a dry filter paper, rejecting the first few portions of the filtrate.

8.2.3.2 Transfer 250 ml of the filtrate to another 500 ml beaker, make just acidic with hydrochloric acid and heat to boiling point. To the boiling solution add drop by drop barium chloride solution with stirring until there is no further precipitation of barium sulphate. Remove from heat and render the contents alkaline with ammonium hydroxide solution, and precipitate the calcium and any excess of barium by adding ammonium carbonate solution until no further visible precipitation occurs, followed by the addition of about 1 ml of ammonium oxalate solution. Cool, to about 20°C, transfer to a 500-ml volumetric flask dilute with water to mark and after thoroughshaking, filter through a dry filter paper, rejecting the first few ml of filtrate.

8.2.3.3 Measure out 100 ml of the filtrate and evaporate to dryness in a silica or heat resistant porcelain basin. Expell the ammonium salts from the residue by gently heating the basin over a low flame, being careful to keep the temperature below that of faint redness. Cool the residue, moisten with concentrated hydrochloric acid, again evaporate to dryness and then ignite at a temperature below redness. Take up the residue with water and filter if necessary, collecting the washings along with the filtrate. Determine potash in the solution by the method described under **8.3.**

8.3 Procedure — Transfer the solution obtained as described under **8.2** into a basin and add about 7 ml of perchloric acid solution. Place the basin on a hot-plate or sand bath and carefully evaporate the contents until dense white fumes are copiously evolved. Cool and dissolve the residue in a little hot water. Add about 1 ml of perchloric acid solution and again concentrate to the fuming stage. Thoroughly cool the residue in the basin and stir in 25 ml of alcohol. Allow the precipitate to settle, then pour the clear liquid through a dry filter paper, draining back the precipitate in the basin, and stir in 20 ml more of alcohol. Allow the precipitate to settle, then pour the clear liquid through the filter paper, draining the precipitate in the basin as completely as possible. The filter paper should not be allowed to dry up. Redissolve the precipitate on the filter paper and that remaining in the basin with hot water, collecting the filtrate and washings in the basin. Add 2 ml of perchloric acid solution to the combined solution and evaporate again to the fuming stage. Cool the residue in the basin and thoroughly stir the contents with 20 ml of alcohol. Allow the precipitate to cool and settle and pour the clear liquid through a dried and weighed gooch or sintered glass crucible, draining the precipitate as completely as possible from the liquid before adding 5 ml of wash solution. Wash the precipitate by decantation with several similar small portions of the wash solution, pouring the washings through the crucible. Transfer the precipitate to the crucible and wash it well with the wash solution until free from acid. Dry the precipitate perchlorate ($KClO_4$) at 100°C and then heat in a muffle furnace maintained at 300 to 350°C for 15 min.

Carry out a blank determination with an identical solution as in **8.3** but without the sample.

Caution — Use carefully, perchloric acid is highly hazardous and is also highly explosive in nature.

8.4 Calculation — Calculate the potash content (as K_2O) by multiplying the mass of the residue (after blank correction) by 0·34.

9. FLAME PHOTOMETRIC METHOD

9.1 Apparatus

9.1.1 *Flame Photometer*

9.1.2 *Ion Exchange Column* — Made from 30 cm length of standard wall glass tubing, 2·5 cm hard diameter; one end is closed by 1-hole rubber stopper through which is inserted a 2-way stopcock or glass tubing connected to rubber (polyethelene) tubing and compressor clamp. Do not let the stopcock tubing protrude above the stopper. Choose the stopper large enough so that there is no space between the stopper vertex and the column wall. Place a glass wool plug in the bottom of the column and fill with an aqueous suspension of the anion exchange resin to a height of 20 cm. Regenerate the resin after 10 successive aliquots have passed through or as advised by the manufacturer of the resin.

9.2 Reagents

9.2.1 *Anion Exchange Resin* — The commercially available material may be used such as REXYN 203 (OH) DUOLITE A-7 or DUOLITE 41. Permutit S-100 or equivalent.

9.2.2 *Sodium Hydroxide Solution* — 5 percent.

9.2.3 *Ditute Nitric Acid* — approximately 5 percent; 1 : 10 (*v/v*).

9.2.4 *Ammonium Oxalate Solution* — containing 40 g in a litre of solution (4 percent).

9.2.5 *Potassium Nitrate or Potassium Chloride* — Recrystallized reagent grade salt (twice from water); dry for 2-3 h at 105°C. The latter is normally used.

9.2.6 *Methyl Red Indicator* — Dissolve 0·2 g of methyl red in 100 ml of rectified spirit.

9.2.7 *Lithium Nitrate* — (free from detectable concentration of potassium).

9.2.8 *Potassium Acid Phthalate* — (pure salt).

9.2.9 *Diammonium Hydrogen Phosphate* — (pure salt).

9.3 Procedure

9.3.1 *Preparation of Resin Column* — Place about 450 g of the resin in a 4-litre beaker and add enough sodium hydroxide solution to completely float the resin (2 to 3 litres) while it is being stirred. Stir for 30 minutes with electric stirrer. Let the resin settle, and decant the sodium hydroxide solution. Repeat treatment with sodium hydroxide solution 2 times more, decanting sodium hydroxide solution after final treatment. Add 2 litres of water to the resin, stir for a few minutes, let the resin settle, and decant the wash water. Repeat 3 to 4 times. The resin is now in the free base form. Regenerate to the nitrate form by treating 3 times with dilute nitric acid (5 percent) using the same technique as with sodium hydroxide solution. Wash the resin until washings reach a *p*H of 2 or above by back washing in column or by stirring and decanting in a large beaker. Store resin under water in stoppered bottles.

9.3.2 *Preparation of Sample Solution*

9.3.2.1 *For mixed fertilizers and potassium-magnesium sulphate* — Weigh 1·505 8 g of the sample into a 250-ml flask (500-ml flask if the sample contains more than 30 percent K_2O), add 100 ml of water, 20 ml of ammonium oxalate soil and boil for 5 or 10 min. Cool, dilute to volume, mix and pass through dry filter (Whatman No. 1 or equivalent).

9.3.2.2 *For potassium chloride and potassium sulphate* — Dissolve 1·505 8 g of the sample in water and dilute to 500 ml. Filter if necessary, using dry filter (Whatman No. 1 or equivalent).

9.3.3 *Preparation of Standard Curve* — Dissolve 1·293 1 g of potassium nitrate (or 0·953 5 g of potassium chloride) in water and dilute to 500 ml (1 000 ppm K). Prepare standard solutions by dilution covering the range 0 to 80 ppm of K at intervals not greater than 10 ppm, adding appropriate amount of lithium nitrate if internal standard instruments is to be used. Prepare standard curve of emission against concentration, adjusting the instrument so that 40 ppm of K gives reading near mid-scale.

9.3.4 *Determination*

9.3.4.1 *For mixed fertilizers, potassium sulphate and potassium magnesium sulphate* — Transfer a 10 ml aliquot of the sample solution to a 250-ml beaker. Add one drop of methyl red indicator solution and neutralize (as necessary) with dilute nitric acid (1 : 10). Adjust the level of water in the column to the top of the resin and transfer the sample aliquot to the column quantitatively. Open the stopcock to give a flow rate of 2 drops per second, collecting the effluent in a 250-ml volumetric flask. Wash the aliquot in the resin with 2 to 3 small portions of water. Collect 50 to 75 ml of the effluent, then open the stopcock and collect an additional 100 ml by pouring water into the column, making certain that the level of water does not fall below the top of the resin bed. Dilute to volume and mix (if internal standard instrument is used, add required amount of lithium nitrate before diluting to volume). Measure emmission at 768 nm. Determine the concentration of potassium (ppm) from the standard curve. Temperature of the standard and the sample solutions shall not differ by more than 2°C.

9.3.4.2 *For potassium chloride* — Proceed as in **9.3.4.1** above but omit neutralization and resin treatment.

TABLE 1 ALIQUOTS AND FACTORS FOR POTASSIUM DETEMINATION

(*Clause* 9.3.4)

PERCENT K_2O EXPECTED (1)	ALIQUOT (2)	FACTOR (3)
2	No dilution	0·050 0
3 to 4	150	0·083 3
5 ,, 6	100	0·125 0
7 ,, 8	75	0 166 7
9 ,, 13	50	0·250 0
14 ,, 15	40	0·312 5
17 ,, 20	30	0·416 7
21 ,, 25	25	0·500 0
26 ,, 30	20	0·625 0
31 ,, 43	15	0·833 3
44 ,, 63	10	1·250

9.3.5 *Calibration of Instrument* — Weigh 1·505 8 g of potassium acid phthalate and transfer to a 250-ml volumetric flask. Add about 0·5 g of diammonium hydrogen phosphate and proceed as in **9.3.2.1** beginning with 'add 125 ml of water.................'. The calculated potash (K_2O) content is 23·0 percent.

9.4 Calculation

a) *For* K_2O *contents up to 30 percent:*

K_2O content, percent by mass

$$= \frac{\text{Concentration of ppm of K read from standard curve}}{2}$$

b) *For* K_2O *contents greater than 30 percent:*

K_2O content, percent by mass

= Concentration of ppm of K read from standard curve.

APPENDIX A

(*Clause* 0.4)

CORRESPONDING REFERENCE TO TEST METHODS IN IS : 6092 (PART 4)-1971 AND IS : 6092 (PART 4)-1985

Test Methods	*Cl Ref in* IS : 6092 (Part 4)-1971	*Cl Ref in* IS : 6092 (Part 4)-1985	*Remarks*
(1)	(2)	(3)	(4)
Methods	5	5	—
Perchloric acid method	6	8	—
Sodium tetraphenylboron volumetric method	7	6	—
Sodium tetraphenylboron (gravimetric method)	—	7	This method has been incorporated in this revised standard
Flame photometric method	8	9	—

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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